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ELECTRODES FOR FUNCTIONAL NEUROMUSCULAR STIMULATION

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Section B: Electrode Design and Fabrication

B.2 Techniques to Attach Lead Wires to Thin-Film Bonding Pads

Abstract

The goal of this sub-project is to investigate conductive epoxy as a means to attach lead wires to thin-film metallic bonding pads. The initial study, which was designed to test the mechanical and electrical properties of the epoxy after being exposed to simulated accelerated in vivo conditions, has been completed. Stainless steel rods were bonded end to end with silver impregnated epoxy and were left at room temperature for 2 days. Control samples were tested after an additional 5-7 days at room temperature and aged samples were tested after an additional 5 days in a 79° C saline bath. For those samples that had been aged, ultimate tensile strengths were decreased by 53.7% compared to controls and resistances were increased by 11.7 kΩ relative to initial values. While these results suggest the epoxy experiences degradative changes upon exposure to simulated in vivo conditions, further testing of the epoxy may need to be performed before an accurate assessment can be made.

Methods

Preliminary discussion of the fabrication of test specimens and the development of the experimental design were reported in a previous progress report (QPR #1). The experimental system of this study consisted of two stainless steel rods joined together at one end by conductive epoxy. This simple system was chosen to avoid the complex joint geometry of the lead wire - bonding pad junction of the cuff electrode.

Solid stainless steel rods were purchased measuring 3.8 cm in length and 0.32 cm in diameter. One end of each of the stainless steel rods was polished to approximate the platinum surface of electrode contacts. The rods were held with the ends kept parallel to the surface of a variable speed grinder and were polished to a final grit of 0.25mm.

A jig was designed and fabricated that could provide a constant and reproducible force, as well as a stable framework for bonding of each sample. This jig provided a force due to gravity of 4.5 Newton per contact area of the rod. During bonding, a Teflon sleeve was to be placed around the epoxied ends of the rods to prevent leakage and to stabilize the system during curing. Initial trials with the Teflon sleeve resulted in inadequate curing of the epoxy. However, trials without the Teflon sleeve resulted in adequate curing, as determined by the adhesion and hardness of the epoxy and so the sleeve was removed from the set-up. Although manufacturer's instructions cited a cure time of 1-1.5 hours, it was observed that the epoxy was fully cured after 2 hours. Therefore, the cure conditions of this study were as follows:

Temperature:	175°C
Time:	2 hours

Because of the force of the jig pushing the epoxy out from between the rods, a

mechanism was needed to provide adequate spacing without significantly altering the amount of contact surface or significantly interfering with the applied force. An insulated stainless steel wire, 40mm in diameter, was placed in between the rods to serve as a spacer.

Following cure, the samples were left at room temperature for two days. They were then divided into two groups, control and experimental. Five samples served as the control group (samples C1-C5) and were left at room temperature for another 5-7 days before being mechanically tested. Five samples served as the experimental group (samples S1-S5) and were immersed in a 79°C saline bath for 5 additional days before being mechanically tested. This elevated temperature soak (5 days at 79°C) artificially aged the samples to 320 days.

The resistance of the epoxied samples was measured with an ohmmeter several times over the course of the week.

Samples were then mechanically tested on an Instron to obtain ultimate tensile strength. Specially-made jigs were used to hold the samples during testing to eliminate any bending moment on the rods caused by the testing apparatus. Following fracture, the contact areas for each sample were measured using the Java image analysis system. These measurements were then used to calculate tensile strengths normalized to contact area.

Fracture surfaces were examined using both light and electron microscopy in an effort to determine failure mode (either cohesive or adhesive).

After completion of testing of the control and saline-aged samples, additional samples were prepared for exposure to dry and ultrapure ion-free water environments, at room and elevated temperatures. As in the first series of experiments, samples were left at room temperature for two days following cure of the epoxy. One sample (sample U1) was then soaked in room temperature ultrapure water for 7 days. Two samples (samples U2 & U3) were soaked in ultrapure water for 3 days at room temperature and then for 4 days at elevated temperature (79°C). The final two samples (samples CD1 & CD2) were left for 1 day in dry room temperature conditions and then for 6 days in dry, 79°C incubation. These samples were measured for resistance several times over the 9 days, were mechanically tested to failure after those 9 days, and their fracture surfaces were analyzed for failure mode.

Results

Resistance

The results of the resistance measurements for test groups are presented in Tables 1 - 4. As can be seen in Table 1, a general increase in resistance was found for control samples. A one-tailed t-test of the mean difference between initial and final measurements revealed the increase in resistance was significant ($p = 0.0083$), averaging 31%.

Table 1: Resistance of Control Group

	Resistance (ohms)				
Time	**C1**	**C2**	**C3**	**C4**	**C5**
Day #0	21.40			14.90	7.10
Day #1		11.50	3.90		8.00
Day #2		12.00			8.00
Day #3	23.40				
Day #4				21.30	9.00
Day #5			4.20		9.30
Day #6		13.60			
Day #7	24.50			21.90	10.50
Day #8			5.30		
Day #9		14.80			
% Increase	14%	29%	36%	47%	48%
Average Increase = 31%					
t-test (1 tail, paired)	p = 0.0082				

Table 2 presents the resistances for the saline-aged group. As shown, the resistances for these samples dramatically increased with aging. This increase was statistically significant, as determined by a one-tailed t-test of the mean difference between initial and final resistance ($p = 0.0285$). The average increase in this sample group, 28200%, was much larger than that in the control group of only 31%.

Table 2: Resistance of Saline Aged Group

	Resistance (ohms)				
Time	**S1**	**S2**	**S3**	**S4**	**S5**
Day #0		56.40	16.30	14.80	75.20
Day #1		65.30	19.50		
Introduced into Saline Bath					
Day #2	45.00	84.00		17.00	
Day #3			19.80		123.80
Day #7	19100.00	19000.00		900.00	
Day #8			800.00		19000.00
% Increase	42344%	33588%	4807%	5981%	25166%
Average Increase = 28210%					
t-test (1 tail, paired) p = 0.0289					

For samples in the groups exposed to ultrapure water environments, resistances fluctuated, as shown in Table 3. Due to the limited numbers of samples, statistical analysis of the results was not performed.

Table 3: Resistance of Water Aged Groups

	Resistance (ohms)		
Time	**U1**	**U2**	**U3**
Day #0	42.20	34.60	22.40
Day #1	46.60	34.10	18.20
Day #2	55.80	34.60	23.00
Exposed to Ultra Pure Water at Room Temp.			
Day #3	85.60	18.00	50.20
Day #4	66.00	19.80	56.00
Day #5	66.50	25.50	56.50
	Room Temp	Elevated Temp	
Day #6	22.70	27.90	61.20
Day #7	14.60	30.30	61.50
Day #8	14.30	32.50	62.00
Day #9	14.10	33.20	60.80

For the two samples exposed to dry, elevated temperature aging, resistances steadily increased, Table 4. The increase for these two samples averaged 61%, however, the small sample population limited further statistical analysis.

Table 4: Resistance of Dry Aged Groups

	Resistance (ohms)	
Time	**CD1**	**CD2**
Day #0	33.10	30.00
Day #1	34.70	33.00
Day #2	35.00	43.20
Day #3	36.10	47.00
Exposed to Dry 79 C		
Day #4	43.00	45.00
Day #5	46.20	45.50
Day #6	48.50	47.10
Day #7	50.20	48.50
Day #8	51.10	49.10
Day #9	52.40	
% Increase	58%	64%
Average Increase = 61%		

Ultimate Tensile Strength

Force at break was measured for all samples mechanically tested on the Instron. Although samples were assumed to be of similar size, contact area was measured for each of the control and saline-soaked samples. This was then used to determine ultimate tensile strengths (UTS) normalized to contact area, which are presented in Table 5. For samples aged in water and dry environments, contact area was not measured and the raw data are presented in Table 6.

The saline-soaked samples exhibited a statistically significant decrease of 54% in normalized UTS compared to control samples (one tailed unpaired t-test, $p = 0.0029$). Figure 1 depicts the normalized UTS values for the five samples of the control group and the five samples of the saline-aged group.

For samples exposed to dry and water environments, UTS data are limited and inconclusive. For water-soaked samples, only one sample in each group was tested. The other sample which had been soaked in ultrapure water for 3 days at room temperature and another 4 days at elevated temperature (sample U2) broke before ever being tested on the Instron. In the dry, elevated temperature exposed samples, the range in UTS (27.7 - 75.2 N) is within the range of un-normalized data from the control and saline-soaked groups. However, without the contact area to normalize this data, it is difficult to compare with the previous groups.

Table 5:
Normalized UTS of Control and Saline Aged Groups

Sample #	UTS (Newton)	Normalized UTS (N/mm^2)
C1	100.5	20.61
C2	52.3	11.07
C3	75.6	15.63
C4	67.1	14.04
C5	79.5	15.97
Average UTS		15.46
S1	58.4	12.06
S2	30.5	6.38
S3	28.3	5.92
S4	26.8	5.63
S5	27.8	5.80
Average UTS		7.16
% Decrease = 54%		
t-test (1 tail, unpaired) p = 0.0029		

Table 6: UTS of Water and Dry Aged Groups

Sample #	UTS (Newton)
U1	43.6
U3	10.2
CD1	75.2
CD2	27.7

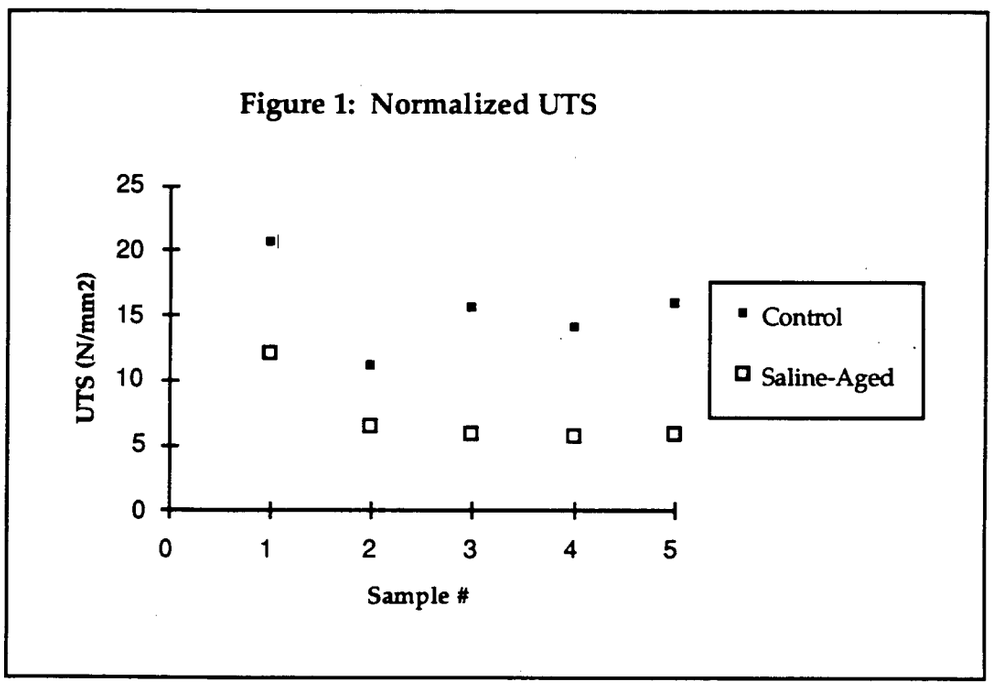

Figure 1: Plot of normalized ultimate tensile strengths (UTS) of both control and saline-aged samples. The saline-aged samples had a statistically significant decrease in UTS relative to controls.

Failure Mode

Fracture surfaces were examined under both light and electron microscopy to determine failure mode. Failures at the epoxy-rod juncture were considered to be adhesive, while failures within the epoxy were considered to be cohesive. All samples had both modes of failure apparent on the fracture surface, as shown in Figures 2 and 3. For control samples and the samples exposed to elevated temperatures in air, the greater proportion of the fracture surface appeared to be cohesive in nature (Fig.2). Alternatively, samples exposed to liquid solutions, both saline and ultrapure water, exhibited more adhesive-type failures (Fig.3).

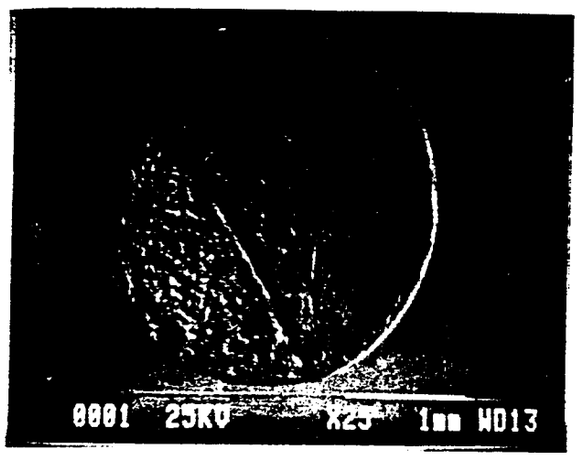

Figure 2: SEM photo of fracture surface of control sample. The predominantly cohesive failures within the epoxy are characterized by the rough textured surface.

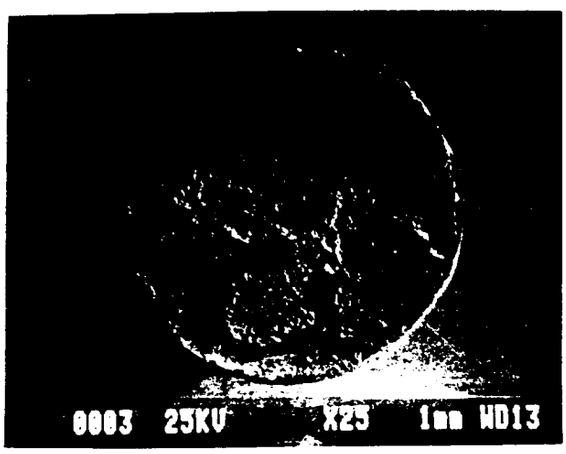

Figure 3: SEM photo of fracture surface of sample aged in saline. The predominantly adhesive failures between the epoxy and the rod are characterized by the smooth surface.

Discussion

The bonded rod system was developed with the intent of providing a simple, reproducible set-up to test the mechanical and electrical response of the epoxy to simulated in vivo conditions. Examination of the results from the control and saline-soaked samples would seem to indicate that the epoxy undergoes unfavorable changes when exposed to those in vivo conditions.

As shown by the data, samples soaked in elevated temperature saline had statistically significant changes in both resistance and UTS. The mechanisms for these changes have not yet been determined. Based on examination of fracture surfaces, it would appear that following soaking, adhesive failures are more likely, indicating that the bond between the epoxy and the metal was most effected by the saline aging. However, the exact mechanism for this change, and whether it was a result of changes in the epoxy or changes in the metal rod, are still unknown.

The cure of the epoxy is of considerable importance in evaluating the results. It was assumed that the epoxy had adequately and completely cured after 2 hours of being pressed in the jigs between the two rods. However, the cure process of the epoxy was not well-supported by documentation from the manufacturer. Exposed surface area, threshold thickness, and other factors may be instrumental in optimally curing the epoxy. A 40μm spacer was placed between each of the rods, but may not have provided the necessary surface area or volume of epoxy for optimal cure.

Examination of the fracture surfaces revealed what appear to be voids in the epoxy along the fracture plane of a few samples. Whether these voids were caused by inherent air bubbles, or by gas evolved during curing is unclear. Equally unclear is the effect these voids may have had on the testing results. As it was only possible to view those voids along the fracture plane, the extent of voids within the remaining volume of epoxy is unknown. Due to the scatter of the data and the few numbers of samples with visible voids, it is especially difficult to assess the possible role of the voids in either the resistance or the tensile strength results.

Also of note is the difference in results between those samples soaked in water and those soaked in saline. The water-soaked samples experienced minor fluctuations in resistance and generally decreased UTS compared to controls, while the saline-soaked samples had a significant increase in resistance and also a significant decrease in UTS. The major difference between these groups was the presence of ions in the saline solution. Because of those ions and because of the dissimilar metal contact, the question of corrosion can be raised. The negative potential of the stainless steel rod relative to the potential of the silver in the epoxy could have led to the creation of a galvanic cell, with subsequent oxidation of the stainless steel and reduction of the silver. The loss of stainless steel at the interface of the silver epoxy and the stainless steel rod could explain both the dramatic increase in resistance, as well as the decreased tensile strength of saline-soaked samples. While metallic corrosion can typically be assessed during high-power electron microscopy evaluation, those high powers were not used during the examination for failure mode. Even at high power magnification, the fracture surface and the epoxy may have obscured any signs of corrosion on the rod. Whether the epoxy has an increased potential for corrosion than the current mode

of electrode contact bonding (spot-welding) has yet to be determined.

Conclusions and Future Work

The results of this study suggest that conductive epoxy proposed for use in attaching lead wires to thin-film bonding pads undergoes degradative changes when exposed to simulated in vivo conditions. However, questions have been raised regarding the experimental design of the study and the cure process of the epoxy. Further testing in a revised experimental design, or simply testing of the epoxy itself, may be initiated before final conclusions regarding the potential in vivo performance of the epoxy will be made.

Section C. Assessment of Electrode Performance in an Animal Model

Section C.1 Quantitative Analysis of Electrode Performance

In previous quarters, acute experiments were conducted on 10 adult cats to quantify the recruitment properties of the 12 contact spiral cuff electrode. A manuscript reporting on the methods used in those experiments was submitted for publication. A copy of that manuscript was provided in QPR #2. In this quarter, analysis of the data from those acute animal experiments was completed. Enclosed is a draft of a manuscript to be submitted to IEEE Transactions in Biomedical Engineering reporting on the input-output properties of the cuff electrode.

Appendix I

Quantification of Recruitment Properties of Multiple Contact Cuff Electrode

Warren M. Grill and J. Thomas Mortimer

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